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SUMMARY

The stress-rupture properties of 5-mil-diameter, type 218CS tungsten wire were determined for rupture times up to about 200 hours at test temperatures from 1200° to 2500° F. Results showed that the rupture properties were superior to those reported for other forms of tungsten, other refractory metals, and superalloys in this temperature range. The onset of primary recrystallization observed between 1800° and 2000° F was indicated by a decrease in microhardness, a decrease in ductility, and a coarsening of grain size. The strength superiority of the wire over the other materials was maintained beyond the onset of primary recrystallization.

INTRODUCTION

In recent years, considerable interest has been generated in the field of fiber-reinforced metallic composites. This interest has been stimulated by the prospect of developing engineering materials that utilize the high strengths available in metallic fibers by using them to reinforce a metallic matrix.

The feasibility of producing composites of this type is demonstrated for a model system in references 1 to 3. These references show that the room-temperature tensile properties of tungsten-fiber-reinforced copper composites are proportional to the tensile properties of the components. Similar results are reported in references 4 and 5, where investigations of the tensile properties of tungsten-fiber-reinforced copper-alloy matrix composites are described. In these investigations the tensile properties of these composites were found to obey a "law-of-mixture" type relation with fiber content. Reference 5 also shows that these relations are valid for elevated-temperature tensile properties of composites.

Although there are many applications for the high tensile property potential of composite materials, there are other applications that require stress for long times at

elevated temperatures. For these applications, the stress-rupture properties of composites must be determined. Since the properties of a composite are dependent on the properties of the components and since the fiber is the primary strengthening component in the composite, the first step toward designing a composite and analyzing its behavior in stress-rupture would be the determination of the stress-rupture properties of the fiber.

Tungsten fibers, which were used in the previous investigations (refs. 1 to 5) represent a logical choice for study because of their high tensile strength at both room and elevated temperatures and because of their elevated-temperature stability. Tensile data for tungsten in rod or bulk form are available over a wide temperature range; however, comparison of the tensile properties of wire (refs. 5 to 8) and rod (refs. 7 to 9) forms indicates that data for tungsten in rod form are considerably lower than data for tungsten in wire form, both at room and elevated temperatures.

Stress-rupture data for rod tungsten are also available over a wide temperature range. In view of the difference, however, in tensile properties between rod and wire, the rod tungsten rupture data may not be applicable to wire. Very few stress-rupture data for tungsten wire are available in the literature. Data presented in reference 10 show the stress-rupture properties of 70-mil-diameter tungsten wire tested at temperatures of 3000° to 4400° F for rupture times up to 200 minutes. These data show that, at the lower test temperatures, wire was slightly better than rod tungsten, but the advantage was lost at the higher test temperatures. Data are presented in reference 11 for the stress-rupture properties of 5-mil-diameter, as-drawn tungsten wire at temperatures of 1500° and 1800° F tested in vacuum for rupture times up to 225 hours. These results are considerably higher than those of bulk tungsten. Since practically no data are available for fine-diameter tungsten wire at temperatures of 1200° to 2500° F, which is a temperature range of interest, it was felt desirable to conduct stress-rupture tests on tungsten wire in these temperature and size ranges.

This investigation was therefore conducted to determine the stress-rupture properties of 5-mil-diameter, as-drawn tungsten wire at test temperatures ranging from 1200° to 2500° F and to compare these results with those of some of the stronger materials for which data are available in this temperature range. Finally, it was intended to determine whether the stress-rupture properties of the tungsten wire were sufficiently high to have promise for use as a reinforcing fiber for high-temperature composites.

The wires were tested in a vacuum of 1×10^{-6} to 5×10^{-5} torr for rupture times up to 225 hours. The fracture area of the wire specimens was examined after fracture and reduction-in-area measurements were made. Microhardness and metallographic examinations were also made.

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MATERIALS

All wire used in this investigation was taken from a single spool of 5-mil-diameter tungsten wire (General Electric type 218CS) in the as-received condition. This type of wire was selected because it permitted comparison of results from previous investigations (refs. 1, 2, 4, 5, and 11) in which the same type of wire was used. The wire was selected for use in the previous work because of its high tensile strength.

Type 218 tungsten wire was made by adding small amounts of potassium silicate and aluminum chloride in aqueous solution to tungsten oxide powder and subsequently reducing the oxide to powdered metal for processing into bar stock by powder metallurgical techniques. The bar was ultimately drawn into wire (ref. 12). Thus, type 218 wire is doped tungsten. This type of wire is generally used at incandescent temperatures and is deliberately processed to yield a large-grained recrystallized structure with proper orientation of grain boundaries with respect to the wire axis when used at these temperatures (ref. 13). This structure is associated with mechanical and thermal stability at temperatures in excess of 4000° F. The CS represents an industrial designation, which indicates that it is the same type of wire as in the as-drawn condition but has received an additional cleaning and straightening operation.

APPARATUS

The equipment used to conduct constant-load stress-rupture tests is shown in figure 1. p. 6

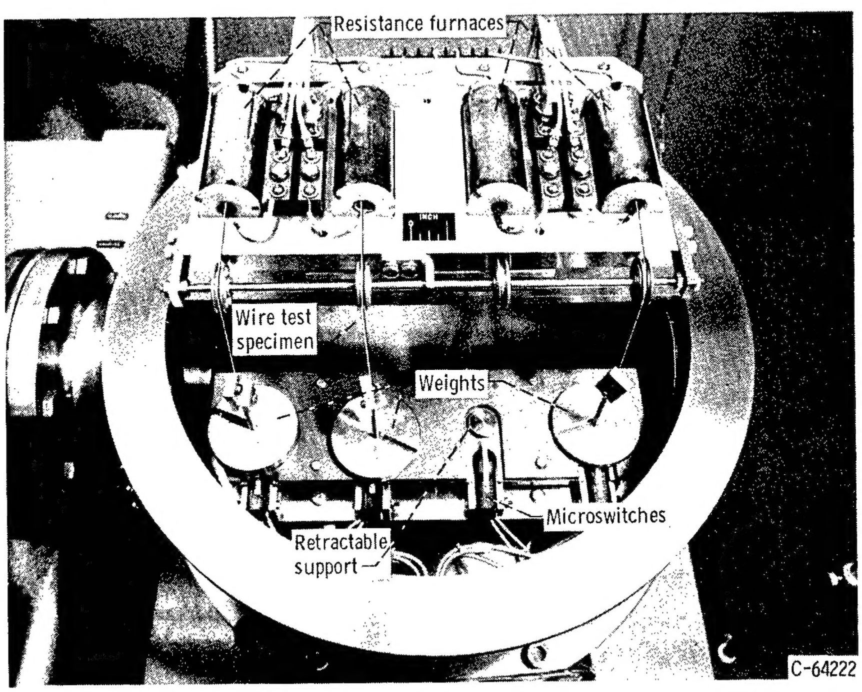

Figure 1. - Filament stress-rupture apparatus showing layout of furnaces, loading train, and microswitches located within environmental chamber.

The wire was cut to 15-inch lengths and threaded through the tantalum-wound resistance furnaces mounted horizontally on a bedplate. The wire test specimen was clamped to a fixed mount, strung through the furnace, passed over a pulley, and attached to the appropriate weight. Test loads were determined on a double pan balance with a sensitivity of ±0.1 gram. The weights were supported by the retractable supports, while the furnaces and wire test specimens were heated to the test temperature and stabilized. Located directly under the weights are microswitches, which are actuated by the fallen weights as each specimen breaks. Each microswitch is connected in series with a furnace and an elapsed time meter. When the specimen failed, the microswitch shuts off the furnace and the elapsed time meter. In addition, some short time tests were timed with a stop watch.

The entire assembly was covered by a glass or a cooled metal bell jar. Testing was conducted in a vacuum of 1×10^{-6} to 5×10^{-5} torr. Individual furnace temperatures were monitored with platinum - platinum-13-percent-rhodium thermocouples. The thermocouples were protected and supported by a two-hole insulating ceramic tube positioned parallel to the long axis of the furnace and parallel to the wire under test. The unprotected thermocouple bead was positioned within 0.5 millimeter of the wire specimen at the midpoint of the hot zone of the furnace. Since the thermocouple bead was not in actual contact with the wire specimen, a calibration was necessary to correlate the temperature of the specimen to that of the monitoring thermocouple. To make this calibration, a butt-welded thermocouple was threaded through the furnace and fixed in the same manner as the test specimen; that is, one insulated leg was passed through the fixed mount, while the other insulated leg was passed over the pulley and attached to the weight pan. The thermocouple was not loaded but was merely attached in a manner so as to avoid slack and maintain position. The butt-welded bead was placed in the center of the hot zone and within 0.5 millimeter of the monitoring thermocouple. The readings of these two thermocouples were then compared as the furnace was heated and after it was allowed to stabilize. The thermocouples read within $\pm3^{\circ}$ F of each other after stabilization.

During the stabilization and testing periods, the monitoring thermocouples were recorded by a recording potentiometer with a sensitivity of $\pm2^{\circ}$ at 1800° F and monitored by a potentiometer with a sensitivity of $\pm0.5^{\circ}$ F. The test temperature did not vary more than $\pm5^{\circ}$ F during the course of a test.

Frictional losses associated with the pulley over which the wire specimen rests were approximately 1 percent of the suspended load. This was determined by measuring the additional weight required to cause movement of the balanced weights suspended over the pulley.

The apparatus and the procedure used to test the filaments in stress-rupture are described in detail in reference 11.

TABLE I. - STRESS-RUPTURE PROPERTIES OF 5-MIL-DIAMETER, AS-DRAWN TUNGSTEN WIRE

Specimen	Temperature, °F	Stress, psi	Life, hr	Area reduction, percent	Room-temperature bend behavior	Cracking
5W-12-01	1200	205 000	0.6	87	Ductile	None
5W-12-02		200 000	.5	83		
5W-12-03		195 999	.9	82		
5W-12-04		190 000	5.0	82		
5W-12-05		185 000	5.0	79		
5W-12-06		185 000	5.4	79		
5W-12-07		180 000	8.6	84		
5W-12-08		175 000	9.9	84		
5W-12-09		170 000	18.1	78		
5W-12-10		165 000	37.1	74		
5W-12-11		160 000	49.2	76		
5W-12-12		160 000	57.6	79		
5W-12-13		155 000	76.5	66		
5W-12-14		153 000	93.6	73		
5W-12-15		148 000	168.0	87		
5W-15-01	1500	200 000	(b)	88	Ductile	None
5W-15-02		175 000	(b)	87		
5W-15-03		175 000	(b)	90		
5W-15-04		150 000	.6	85		
5W-15-05		150 000	.9	87		
5W-15-06		150 000	1.1	86		
5W-15-07		145 000	1.8	86		
5W-15-08		140 000	1.2	87		
5W-15-09		140 000	1.4	83		
5W-15-10		135 000	3.4	85		
5W-15-11		130 000	6.5	85		
5W-15-12		125 000	16.1	86		
5W-15-13		120 000	63.5	87		
5W-15-14		115 000	192.5	86		
5W-18-01	1800	150 000	(b)	90	Ductile	None
5W-18-02		135 000	.1	95		
5W-18-03		125 000	.2	92		
5W-18-04		120 000	.24	90		
5W-18-05		115 000	.7	93		
5W-18-06		110 000	3.0	93		
5W-18-07		110 000	4.0	87		
5W-18-08		107 000	7.1	90	Brittle	
5W-18-09		105 000	11.0	90		
5W-18-10		105 000	19.0	86		
5W-18-11		100 000	7.6	88		
5W-18-12		100 000	20.5	79		Thin
5W-18-13		97 000	79.8	82		Thin
5W-18-14		95 000	95.9	77		Thin
5W-18-15		90 000	224.7	70		Thick
5W-20-01	2000	100 000	0.37	90	Brittle	None
5W-20-02		95 000	.66	90		None
5W-20-03		90 000	.8	60		Thin
5W-20-04		85 000	2.2	63		
5W-20-05		80 000	2.0	48		
5W-20-06		80 000	2.6	64		
5W-20-07		80 000	2.7	56		
5W-20-08		75 000	3.2	48		
5W-20-09		75 000	7.5	59		
5W-20-10		75 000	7.7	59		Thick
5W-20-11		72 000	9.7	44		Thick
5W-20-12		72 000	9.8	41		Thin
5W-20-13		72 000	15.6	12		Thin
5W-20-14		70 000	15.1	12		Thin
5W-20-15		70 000	17.1	(a)		(a)
5W-20-16		70 000	22.8	23		Thin
5W-20-17		65 000	69.5	(a)		Thin
5W-20-18		60 000	146.8	19		Thick
5W-23-01	2300	63 000	1.0	24	Ductile	Thin
5W-23-02		60 000	1.5	34	Ductile	Thin
5W-23-03		55 000	4.5	54	Brittle	None
5W-23-04		50 000	7.8	45		None
5W-23-05		50 000	10.8	19		Thick
5W-23-06		45 000	11.1	36		Thin
5W-23-07		45 000	24.4	27		Thin
5W-23-08		40 000	76.4	15		Thin
5W-23-09		38 000	107.0	29		Thick
5W-25-01	2500	50 000	.8	28	Ductile	Thin
5W-25-02		45 000	2.0	50	Ductile	Thin
5W-25-03		42 000	2.5	42	Brittle	None
5W-25-04		40 000	5.2	50		
5W-25-05		40 000	6.6	45		
5W-25-06		37 000	6.6	52		
5W-25-07		35 000	13.8	44		
5W-25-08		35 000	16.6	54	Ductile	
5W-25-09		30 000	24.9	27		
5W-25-10		30 000	35.1	23		
5W-25-11		25 000	116.8	33		
5W-25-12		25 000	162.4	28	Brittle	Thin

[a]No data available.
[b]Broke on loading.

PROCEDURE

Ductility

Elevated temperature. After testing, the fracture area of each wire specimen was examined on a microscope comparator at a magnification of 100, and reduction-in-area calculations were made. The initial diameter was 0.0050±0.00005 inch. The final diameter at fracture was measured with a comparator micrometer that had a sensitivity of ±0.00005 inch. The reduction-in-area values were considered accurate to ±1-percent reduction in area. The reduction-in-area data were, in most cases, the average of two observations. Along with reduction-in-area measurement, it was noted whether or not circumferential cracking was observed near the fracture.

Room temperature. The room-temperature ductility was determined after rupture failure by a simple bend test. The specimen wire was manually bent near the fracture edge through a 90° arc over a 25-mil mandrel (a 5t bend test). Wires that were bent without failure were classified as ductile, while those that fractured were classified as brittle.

Metallographic Examination

After testing, wires were mounted and polished for metallographic examination. The wires were etched with Murakami's Etchant (10 cc potassium hydroxide, 10 cc potassium ferricyanide, and 100 cc water) to reveal the structure of the tungsten wire. Photomicrographs were then taken at magnifications of 250 and 1000.

Microhardness Testing

Microhardness tests were conducted within 1/4 inch of the fracture edge on polished longitudinal sections of tungsten wire. Diamond Pyramid hardness of wire in metallographic mounts was determined with a 200-gram load on the indentor.

RESULTS

Stress-Rupture Results

Stress-rupture data for 5-mil-diameter, as-drawn tungsten wire are shown in table I and plotted on a log stress - log rupture time basis in figure 2. Tests were conducted

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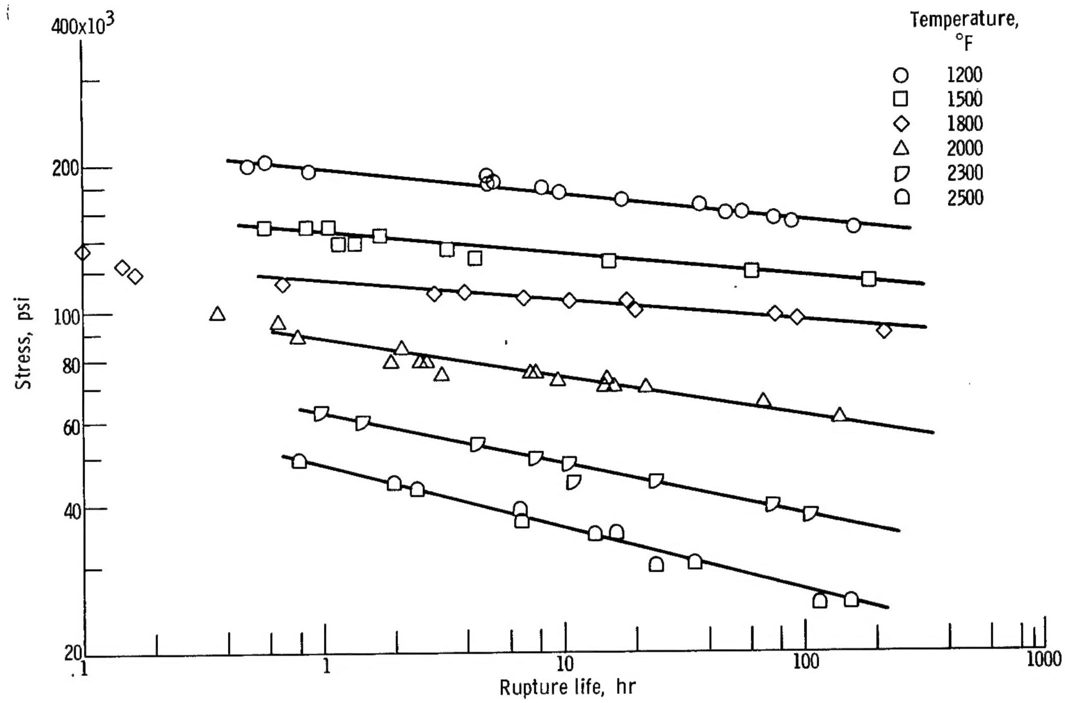

Figure 2. - Stress as function of rupture life for 5-mil-diameter, as-drawn tungsten wire.

TABLE II. - CALCULATED STRESS[a] TO GIVE RUPTURE TIMES OF 1, 10, AND 100 HOURS FOR 5-MIL-DIAMETER, AS DRAWN TUNGSTEN WIRE

Temperature, °F	Stress for rupture, psi		
	1 hr	10 hr	100 hr
1200	199 100	175 500	154 800
1500	145 600	130 400	116 700
1800	116 000	104 500	94 200
2000	88 100	73 300	61 000
2300	62 800	49 000	38 300
2500	49 100	35 900	26 200

[a]Calculated from values which yielded least-squares fit for the equation log stress = log a + slope × log rupture time where a is stress to give rupture time of 1 hr.

over a temperature range from 1200° to 2500° F, with stresses selected to cause failure in times up to about 200 hours.

The lines shown in figure 2 represent a least squares fit of data for each test temperature. There was little scatter, and the data remained linear with test times up to about 200 hours for each temperature. No break or discontinuity in the curves was observed up to these failure times. The curves for each temperature are relatively flat, and a small change in stress caused a large difference in rupture time. The curves were also nearly parallel, but the slope became slightly steeper at the higher test temperatures.

The results of a least squares fit through the data at each test temperature are tabulated in table II. The equations of the lines obtained at each temperature were solved for the stress intercept to cause rupture at 1, 10, and 100 hours.

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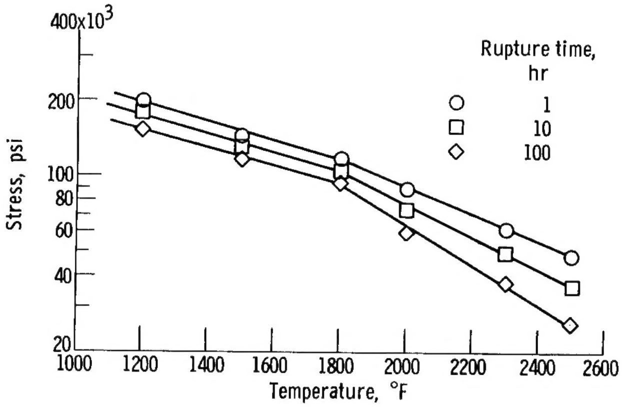

Figure 3. - Stresses required to give rupture lives of 1, 10, and 100 hours for 5-mil-diameter, as-drawn tungsten wire at various temperatures.

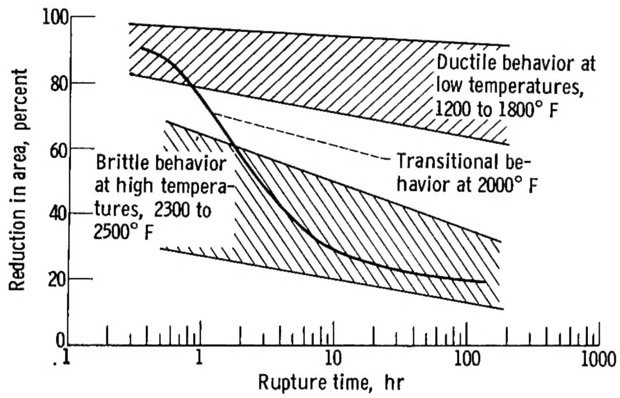

Figure 4. - Changes in ductility with test temperature for 5-mil-diameter, as-drawn tungsten wire.

The data shown in table II were plotted in figure 3 as a function of temperature. This curve shows the relation between test temperature and the log of stress required to cause rupture in 1, 10, and 100 hours. A linear decrease in the log of stress for rupture for each of the times was obtained between 1200° and 1800° F. A second linear decrease with a steeper slope was obtained for each time curve for test temperatures between 1800° and 2500° F. The curves for each of the selected rupture times showed this discontinuity or breakoff between 1800° and 2000° F. The lower temperature portions of the curves for different rupture times were parallel; at the higher-temperature portion, however, the curves were divergent. The slope became greater with increasing rupture time. The divergence of the higher-temperature portion of these curves indicated that increased time at these elevated temperatures had an additive effect beyond that of increased temperature alone in causing property degradation.

Ductility Results

Elevated-temperature ductility. - The elevated-temperature ductility of the tungsten wire specimens tested in this investigation was determined through reduction-in-area measurements. The reduction-in-area results are presented in table I. A plot of reduction in area against log rupture time is presented in figure 4. The data in the table for specimens tested at 1200°, 1500°, and 1800° F exhibited high reduction-in-area values and were combined into a single scatter band in figure 4. The data for specimens tested at 2300° and 2500° F showed low reduction-in-area values and were combined into another scatter band in figure 4. The ductility of specimens tested at 2000° F showed a transitional-type behavior. At short rupture times the ductility was within the range of

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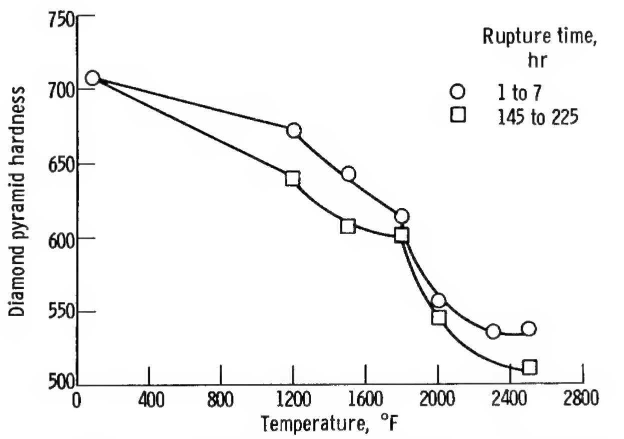

Figure 5. - Microhardness of 5-mil-diameter, as-drawn tungsten wire after short and long rupture times at temperatures up to 2500° F.

the 1200° to 1800° F scatter band, while at intermediate rupture times the ductility fell to between the two scatter bands, and at longer rupture times the ductility fell within the 2300° to 2500° F scatter band. In general, both scatter bands showed reduced ductility with increasing rupture time.

Room-temperature ductility. - The room-temperature ductility of the tungsten wire specimens tested in this investigation was determined through a bend test at room temperature following stress-rupture failure. The bend behavior of these specimens is shown in table I. Wire specimens tested for short and long times at 1200° and 1500° and for short times at 1800° F were ductile at room temperature. Brittle failures were encountered after longer rupture times at 1800° F. Wires tested at 2000°, 2300°, and 2500° F were generally brittle and fractured on bending. The room-temperature ductility decreased with increasing time under test at temperatures of 1800° F and above.

Microhardness Results

The results of Diamond Pyramid hardness tests, using a 200-gram load, taken on wire specimens are shown in figure 5. Two curves are shown in this figure, one for short rupture times (1 to 7 hr) and another for longer rupture times (145 to 225 hr). Both curves show a drop in hardness from the as-received condition with increasing test temperature. Both curves also show a pronounced drop in hardness at test temperatures above 1800° F. Slightly lower hardness values were obtained from wire specimens with longer rupture times when compared with specimens with short failure times. Each hardness value shown in figure 5 represents the average of at least two hardness readings.

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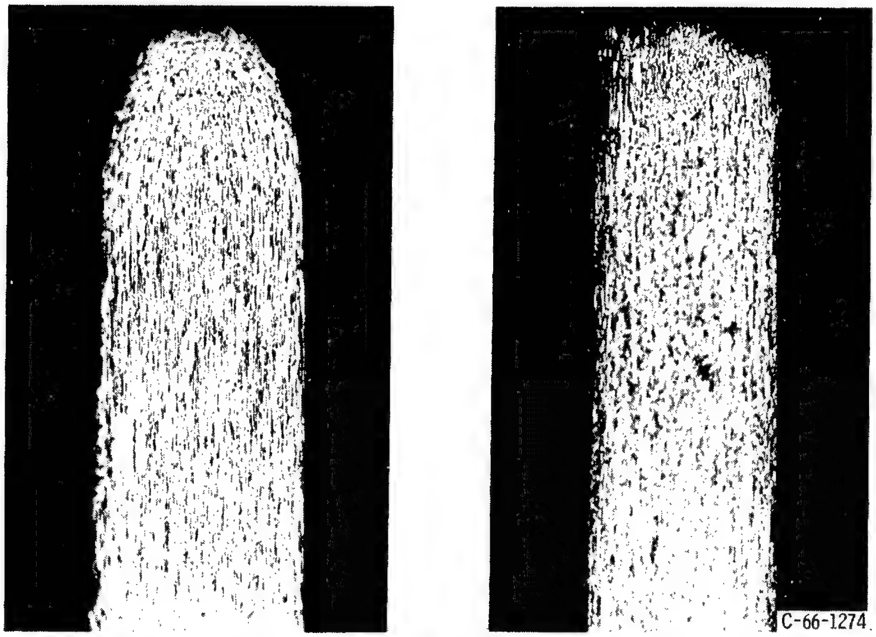
Metallographic Results

Photographs of fractures typical of the ductile lower temperature behavior and the brittle higher temperature behavior are shown in figure 6.



(a) Ductile; specimen 5W-12-05.

(b) Brittle; specimen 5W-25-11.

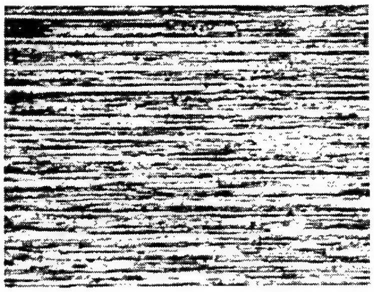
Figure 6. - Photomicrographs of typical ductile and brittle fracture modes. Murakami's etch; X250.

Photomicrographs of the microstructures of typical specimens that failed at rupture times up to about 16 hours at various temperatures are shown in figure 7. After testing at temperatures up to 1800° F, the microstructure shows a fibrous structure typical of the as-drawn wire. These photographs show that the microstructure undergoes some coarsening after exposure to increasing test temperatures. There is not much change in microstructure with exposure up to 1800° F. Exposure to test temperatures of 2000° F and above, however, showed a much greater amount of coarsening of grains, and at 2500° F much of the fibrous structure had been lost. At these higher temperatures, it appears that the fibrous structure is being replaced with a columnar grain structure, which maintains the same general orientation but has a coarser appearance than the fibrous structure.

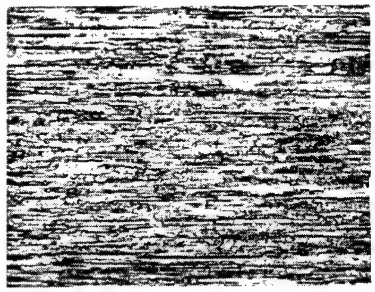
In addition to the microstructural changes, some wire specimens exhibited circumferential cracking. The occurrence of cracking is noted in table I, and a photograph of a typical cracked specimen is shown in figure 8.

DISCUSSION

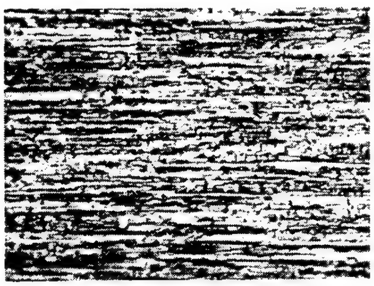
The stress-rupture properties obtained for 5-mil-diameter tungsten wire in this investigation were found to compare favorably with other materials under consideration for use in this temperature range. Stress-rupture properties of tungsten wire were found to



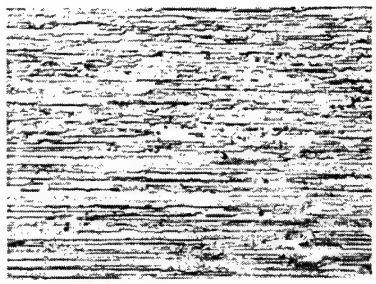
(a) As received; no exposure.



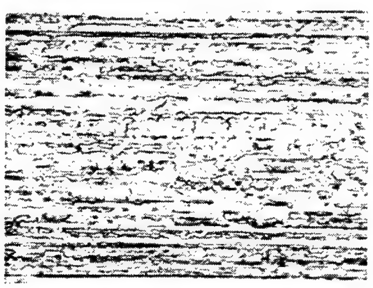
(b) Specimen 5W-12-05; temperature, 1200° F, exposure, 5.0 hours.



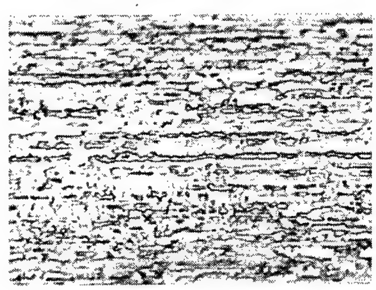
(c) Specimen 5W-15-11; temperature, 1500° F; exposure, 6.5 hours.



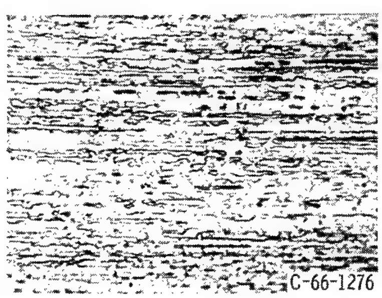
(d) Specimen 5W-18-08; temperature, 1800° F; exposure, 7.1 hours.



(e) Specimen 5W-20-14; temperature, 2000° F; exposure, 15.1 hours.



(f) Specimen 5W-23-04; temperature, 2300° F; exposure, 7.8 hours.



(g) Specimen 5W-25-08; temperature, 2500° F; exposure, 16.6 hours.

Figure 7. - Photomicrographs of tungsten wires tested at various temperatures. Murakami's etch; X1000 (reduced 40 percent in printing).

Figure 8. - Photomicrograph of fracture zone of tungsten wire showing circumferential cracking on surface. Specimen 5W-18-15; Murakami's etch; X1000 (reduced 13 percent in printing).

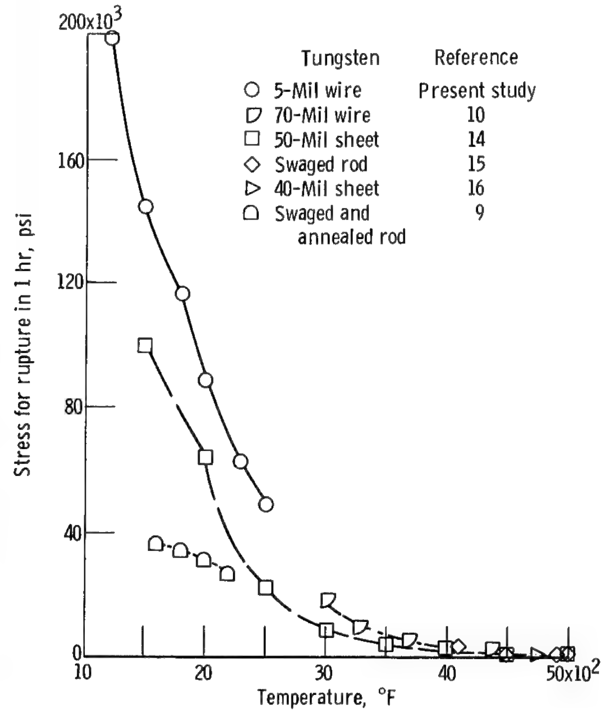


Figure 9. - Stress required for rupture in 1 hour as function of temperature for several forms of tungsten.

be superior to published data available for other forms of tungsten, other refractory metals, and superalloys. The properties obtained were related, to a large degree, to the recrystallization behavior of the tungsten wire.

Comparison of Stress-Rupture Properties of Tungsten Wire With Other Forms of Tungsten

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The stress-rupture results of 5-mil-diameter tungsten wire are compared with results from other studies of the stress-rupture properties of tungsten, in different forms, as a function of temperature in figure 9. The plot covers the temperature range from 1000° to 5000° F and shows the stress to cause rupture in 1 hour. The 1-hour rupture stress was used since, in many cases, only short-time rupture data were available in the literature. These data are plotted on a linear plot to show more clearly the differences in properties. The data for rod tungsten from reference 9 were extrapolated to 1-hour lives by using the Larson-Miller parameter plot given in the reference. The rest of the data presented in figure 9 were taken directly from references 10 and 14 to 16.

Figure 9 shows that, at temperatures up to 2500° F, the as-drawn tungsten wires tested in this investigation had properties better than those of the 50-mil-thick, as-received tungsten sheet reported in reference 14 and also the 60-mil sheet (ref. 17) that had about the same properties (not plotted). Data for tungsten rod, annealed for 1 hour at 2900° F (ref. 9), are also included for comparison. In this temperature range, up to 2500° F, the 1-hour rupture stress for tungsten wire is about $1\frac{1}{2}$ times that of tungsten sheet reported in reference 14 and about 3 to 4 times that of recrystallized tungsten rod annealed for 1 hour at 2900° F reported in reference 9.

The plot also shows that the superior properties reported in the literature for highly worked tungsten, in wire or sheet form, are lost in the 3000° to 5000° F temperature range. The merging of the data for wire, sheet, and rod above 3000° F is believed to result from recrystallization. It would be expected that the data from wires tested in this investigation also would merge at these higher temperatures.

Comparison of Stress-Rupture Properties of Tungsten Wire With Superalloys and Other Refractory Metals and Alloys

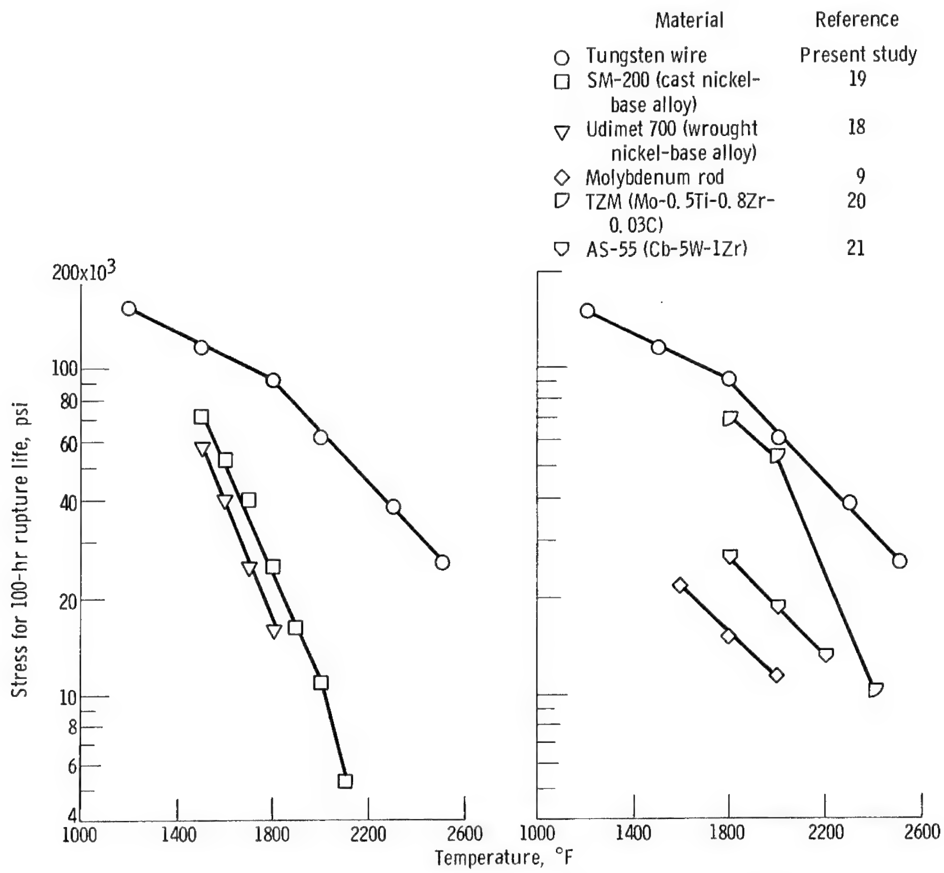
p. 15

A great deal of research is being directed toward the creation of improved structural materials for use in the 1500° to 2500° F temperature range. Nickel- and cobalt-base superalloys are usually used for applications in the lower temperature portion of this range. Figure 10(a) shows a comparison of the 100-hour rupture strengths of tungsten wire and two of the better nickel-base superalloys currently available, Udimet 700 (wrought) (ref. 18) and SM-200 (cast) (ref. 19). The tungsten wire studied in this investigation exhibited rupture strengths that were superior to those of the superalloys throughout the entire temperature range considered.

The tungsten wire also exhibited a much greater thermal stability, as indicated by the slopes of the curves, at these temperatures. Furthermore, the strength of superalloys decreased rapidly with increased temperature above 2100° F. In contrast, the 100-hour rupture strength for the tungsten wire decreased less rapidly with increasing temperature and showed an even greater superiority over superalloys at higher temperatures, as shown in figure 10(a).

Tungsten has a density about twice that of these superalloys; thus, where strength/density ratio is important, the strength advantage of tungsten wire is reduced. However, at 2100° F, tungsten remains stronger by a factor of 5 when stress for 100-hour rupture-life/density ratio values are compared.

Refractory metals and alloys are generally used for high-stress applications at temperatures above 2000° F, since this temperature range is beyond the normal application



(a) Tungsten wire and two nickel-base superalloys.

(b) Tungsten wire and molybdenum and columbium alloys.

Figure 10. - Stress required for rupture in 100 hours as function of temperature.

range of superalloys. Figure 10(b) compares the stress for rupture in 100 hours of tungsten wire with molybdenum (ref. 9), an arc-cast, stress-relieved molybdenum alloy TZM (ref. 20), and a columbium-base alloy AS-55 (ref. 21). The tungsten wire is stronger than all other materials shown in figure 10(b). It was shown previously (refs. 1 to 5) that the tensile properties of a composite are proportional to the properties of the components. It may be assumed that in stress-rupture testing a similar relation would be valid, and since the fiber is the primary strengthening component, the strongest fiber would be expected to result in composites having the highest rupture strength. This investigation has shown that the stress-rupture properties of tungsten wire are superior to those of currently available superalloys, other forms of tungsten, and other refractory metals at temperatures up to 2500° F. Therefore, it appears that tungsten wire has promise for use as a reinforcing fiber for composites in this temperature range.

It is also significant to note the increase in rupture strength of the molybdenum alloy TZM compared with unalloyed molybdenum. The alloy is about four times stronger. If a tungsten alloy with a comparable strength increase were drawn into wire form, greatly

superior properties could be obtained. There has been little demand for tungsten alloys with attractive stress-rupture properties in this temperature range (1500° to 2500° F). Most of the research on tungsten alloy development has been in the 3000° to 5000° F temperature range. In view of the superior properties of tungsten wire from 1200° to 2500° F, it would appear very worthwhile to devote research effort toward the development of tungsten-base alloys specifically developed for use as reinforcement of composites in this lower temperature range.

Effect of Recrystallization on the Life of Tungsten Wire in Stress-Rupture

p. 16

The curves for each of the rupture times in figure 3 (p. 8) showed a discontinuity or breakoff between 1800° and 2000° F. In addition, there was a divergence of the curves for different rupture times in the temperature region from 1800° to 2500° F.

This divergence of rupture data at these higher temperatures was accompanied by a reduction in ductility, as evidenced by a lowering of reduction-in-area values during test and a brittle room-temperature bend behavior after testing. These changes in properties might be expected to be caused by the onset of recrystallization of the tungsten wire. An attempt will be made to relate the recrystallization behavior of the wire to the rupture life in this section, while the relation to ductility will be discussed in the final section of the discussion.

Analysis of metallographic (fig. 7) and microhardness data (fig. 5) suggested the presence of recrystallization. Between 1200° and 1800° F, there was a gradual coarsening of the fibrous structure of the wire, with an accompanying reduction in hardness. Between 1800° and 2000° F, there was a significant drop in Diamond Pyramid hardness (610 to 550). For specimens tested at temperatures between 2000° and 2500° F, there was a further drop in Diamond Pyramid hardness (550 to 525) accompanied by an increasing amount of replacement of fibrous grains by columnar grains. The microhardness of all wire specimens decreased with increasing time at a given temperature.

Similar changes in microstructure and microhardness have been observed by other investigators. It has been reported (refs. 22 and 23) that, below a temperature of about 1500° F, recovery processes were present with no accompanying change in microstructure. Between about 1500° and 3000° F, there was a coarsening of the fibrous structure, with the replacement of fibrous grains by columnar grains. This latter process is considered a subgrain growth by references 22 and 23 and termed primary recrystallization by reference 22. Above 3000° F, equiaxed grains are formed, which both authors call secondary recrystallization. Reference 22 reports that the room-temperature tensile strength

of tungsten wires was reduced somewhat by primary recrystallization in this temperature range; however, the decrease was not nearly as great as that observed for wires that had been given annealing treatments to cause secondary recrystallization. Thus from these considerations, it would appear that the microstructural and microhardness changes observed in the tungsten wire studied in this investigation indicated the onset of recrystallization at temperatures between 1800° and 2000° F. It was also noted that a Diamond Pyramid hardness of 335 was obtained on a wire specimen that had been fully recrystallized to a very large equiaxed grain structure by heating to about 5000° F. This hardness was significantly below the 500 to 550 observed for the recrystallization processes encountered over the 1800° to 2500° F temperature range during stress-rupture testing. Equiaxed grain growth did not occur in this temperature range, and the microstructure was a heterogeneous mixture of fibrous and columnar grains.

In view of the hardness range resulting from stress-rupture testing of the tungsten wires tested in this temperature range and in view of the coarsening of the microstructure caused by the replacement of fibrous grains by columnar grains, it would appear that the recrystallization observed was similar to that observed in references 22 and 23 and termed primary recrystallization by reference 22. The designation of this recrystallization process as primary recrystallization will also be used in this report. It would also appear that the deviations in stress-rupture properties observed in the 1800° to 2000° F temperature range and above were caused by the onset of primary recrystallization. It was reported in reference 9 that a change in the slope of the plot of the log minimum creep rate against temperature was noted at these same temperatures. A discontinuity of the Larson-Miller parameter plot also was observed.

Although the primary recrystallization observed in the tungsten wires tested in stress rupture in this investigation reduced the stress-rupture life of the wires, it should be emphasized that the decrease in rupture properties was slight. The replacement of the fibrous grains by the columnar grains to form a heterogeneous microstructure of fibrous and columnar grains was associated with the retention of a great portion of the strength of the material.

Effect of Recrystallization on Ductility of Tungsten Wire in Stress-Rupture

The preceding discussion showed the role of primary recrystallization and its effect on the stress-rupture life of tungsten wire. The primary recrystallization that occurred during stress-rupture testing between 1800° and 2500° F caused only a slight reduction in life, but it had a much greater effect on the ductility.

The tungsten wires were brittle at room temperature after exposures for long times at 1800° F, and this was generally true for all specimens tested at 2000° F and above. This indicated that the onset of primary recrystallization was associated with an increase in the ductile-to-brittle transition temperature of the wires to above room temperature.

The elevated-temperature ductility of wires tested in stress-rupture, as indicated by reduction-in-area measurements, was also associated with primary recrystallization. The reduction in area of the tungsten wires exhibited high values at temperatures from 1200° to 1800° F, a transition at 2000° F, and low values from 2300° to 2500° F. These results are presented in figure 4 (p. 8).

The reduced ductility of the tungsten wires of this investigation appears to be anomolous to that indicated in the literature for other forms of tungsten in this temperature range. Data obtained in this temperature range for swaged and annealed tungsten rod (ref. 9) and for 50-mil tungsten sheet (ref. 14) showed increasing ductility with increasing temperature within the 1200° to 2500° F temperature range. The swaged and annealed rod exhibited greater ductility than the sheet. In addition, high ductility was observed at 2500° F in references 17 and 24.

Several factors should be taken into consideration in analyzing this apparent anomoly. The behavior of highly worked products, such as tungsten wire or sheet, might be expected to differ from that of swaged tungsten rod. Thus, the high elongation ductility reported in reference 9 might be expected to be more typical of tungsten in rod form, but not in wire or sheet form.

A better basis for comparison of ductility may be to compare the properties of the 218CS tungsten wires tested in this investigation with those of the tungsten sheet reported in reference 14. This allows a comparison of a highly worked, doped form of tungsten (wire) with a less heavily worked form (sheet), which presumably was not doped.

It is possible that the change in ductility behavior, as indicated by decreased reduction of area values and the occurrence of circumferential cracking, could be related to the differences in the amount of mechanical working and the amount of residual doping elements between the wire and the sheet.

The behavior of more highly worked, doped, powder-metallurgy tungsten wire might be expected to differ from that of less highly worked, undoped, powder-metallurgy tungsten sheet. The recrystallization behavior of the wire also would be expected to differ from that of the sheet in that there would be a greater tendency for preferentially oriented recrystallization to form a heterogeneous fibrous-columnar structure. This recrystallization behavior is associated with the deliberate addition of doping impurities. These additions are useful in resisting sag of the wire for their intended use as lamp filaments and in promoting the type of structure in the wire which is associated with increased creep resistance at incandescent temperatures. When used at lower temperatures, how-

ever, these characteristics might influence the ductility behavior of the wire. The ductility of the wire, in worked form with a fibrous grain structure, is good compared with wrought tungsten sheet. Good ductility might be obtained if the impurities were dispersed throughout the structure, which would be expected to result from the extensive wire-drawing process. The onset of recrystallization, as shown by the formation of columnar grains, might upset this uniform distribution of impurities. Recrystallization of the grains may sweep these impurities to the grain boundaries in a manner similar to that suggested by reference 25. The concentration of these impurities at the grain boundaries might be expected to affect the ductility seriously.

CONCLUSIONS

The stress-rupture properties of 5-mil-diameter tungsten wire were determined for rupture times up to about 200 hours and for temperatures from 1200° to 2500° F. Rupture properties were correlated to microstructure, microhardness, and ductility. The results obtained led to the following conclusions:

1. The stress levels that were maintained for long rupture times were superior to those reported for other forms of tungsten, other refractory metals, and superalloys. The superior properties indicated that the wire showed promise as a potential fiber reinforcement in the 1200° to 2500° F temperature range.

2. Decreases in microhardness, reduction-in-area ductility, and a coarsening of grain size were observed at temperatures between 1800° and 2000° F. These changes indicated the onset of primary recrystallization. Changes observed in rupture life above these temperatures were also related to primary recrystallization.

3. The strength superiority of the wires over the other forms of tungsten was maintained to the highest temperature tested (2500° F), which was well beyond the onset of primary recrystallization.

Lewis Research Center, and
National Aeronautics and Space Administration,
Cleveland, Ohio, March 17, 1966.

REFERENCES

1. Jech, R. W.; McDanels, D. L.; and Weeton, J. W.: Fiber Reinforced Metallic Composites. Composite Materials and Composite Structures. Proceedings of the Sixth Sagamore Ordnance Materials Research Conference. Rep. MET 661-601, Syracuse Univ. Res. Inst., 1959, pp. 116-143.

2. McDanels, David L.; Jech, Robert W.; and Weeton, John W.: Stress-Strain Behavior of Tungsten-Fiber-Reinforced Copper Composites. NASA TN D-1881, 1963.

3. Kelly, A.; and Tyson, W. R.: Fibre-Strengthened Materials. High-Strength Materials, 2nd International Materials Conference, Berkeley, Calif., V. F. Zackay, ed., John Wiley and Sons, Inc., 1965, pp. 578-602.

4. Petrasek, Donald W.; and Weeton, John W.: Alloying Effects on Tungsten-Fiber-Reinforced Copper-Alloy or High-Temperature-Alloy Matrix Composites. NASA TN D-1568, 1963.

5. Petrasek, Donald W.: Elevated-Temperature Tensile Properties of Alloyed Tungsten Fiber Composites. NASA TN D-3073, 1965.

6. Anon.: Tungsten. Metallwork Plansee, Reutte, Tyrol, Austria, 1959.

7. Lyman, Taylor, ed.: Properties and Selection of Metals, vol. 1 of Metals Handbook, Eighth Ed., American Soc. for Metals, 1961, pp. 1225-1226.

8. Agte, Curt; and Vacek, Jiri: Tungsten and Molybdenum. NASA TT F-135, 1963.

9. Pugh, J. W.: Tensile and Creep Properties of Tungsten at Elevated Temperatures. Proc. ASTM, vol. 57, 1957, pp. 906-916.

10. Hum, Jack K. Y.; and Donlevy, Al: Some Stress-Rupture Properties of Columbium, Molybdenum, Tantalum, and Tungsten Metals and Alloys Between 2400° and 5000° F. Repr. No. 354D, SAE, 1961.

11. Jech, R. W.; Springborn, R. H.; and McDanels, D. L.: Apparatus for Stress-Rupture Testing of Filaments in a Controlled Environment. Rev. Sci. Instr., vol. 35, no. 3, Mar. 1964, pp. 314-315.

12. Swalin, R. A.; and Geisler, A. H.: The Recrystallization Process in Tungsten as Influenced by Impurities. J. Inst. Metals, vol. 86, Nov. 1957, pp. 129-134.

13. Wretblad, P. E.: Manufacture of Tungsten Metal. Powder Metallurgy, J. Wulff, ed., ASM, 1942, pp. 420-435.

14. Drury, C. A.; Kay, R. C.; Bennett, A.; and Albom, M. J.: Mechanical Properties of Wrought Tungsten. (Rep. No. ASD-TDR-63-585, vol. 2, DDC No. AD 427128), Marquardt Corp., Nov. 1963.

15. Green, Walter V.: Short-Time Creep-Rupture Behavior of Tungsten at 2250° to 2800° C. Trans AIME, vol. 215, no. 6, Dec. 1959, pp. 1057-1060.

16. Sutherland, E. C.; and Klopp, William D.: Observations of Properties of Sintered Wrought Tungsten Sheet at Very High Temperatures. NASA TN D-1310, 1963.

17. Tavernelli, J. F.: The Creep and Stress-Rupture Properties of Tungsten Sheet Between 2500° and 4000° F. Rep. No. 64-LMC-226, General Electric Co., Sept. 8, 1964.

18. Lund, C. H.: The Use of Nickel-Base Alloys in the Rotating Parts of Gas Turbines for Aerospace Applications. DMIC Memorandum 145, Defense Metals Information Center, Battelle Memorial Institute, Jan. 1962.

19. Anon.: SM200 - High Temperature Nickel Base Superalloy. Martin Metals Co., Wheeling, Illinois.

20. Schmidt, F. F.; and Ogden, H. R.: The Engineering Properties of Molybdenum and Molybdenum Alloys. DMIC Rep. No. 190 (DDC No. AD 426264), Defense Metals Information Center, Battelle Memorial Institute, Sept. 1963.

21. Schmidt, F. F.; and Ogden, H. R.: The Engineering Properties of Columbium and Columbium Alloys. DMIC Rep. No. 188 (DDC No. AD 426255), Defense Metals Information Center, Battelle Memorial Institute, Sept. 1963.

22. Davis, G. L.: Recrystallisation of Tungsten Wires. Metallurgia, vol. 58, no. 348, Oct. 1958, pp. 177-184; also vol. 58, no. 349, Nov. 1958, pp. 228-232.

23. Pugh, J. W.: The Recovery and Recrystallization of Tungsten. Third Plansee Seminar: De re Metallica, F. Benesovsky, ed., Pergamon Press, 1959, pp. 97-107.

24. Sikora, Paul F.: High Temperature Tensile and Stress-Rupture Properties of Some Alloys in the Tungsten-Molybdenum System. NASA TN D-1087, 1962.

25. Mannerkoski, M.: Recrystallization of "Doped" Tungsten Wire. J. Inst. Metals, vol. 88, May 1960, pp. 397-398.

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